

## Supplementary Material

### Chemical Nature of Spent Coffee Grounds and Husks

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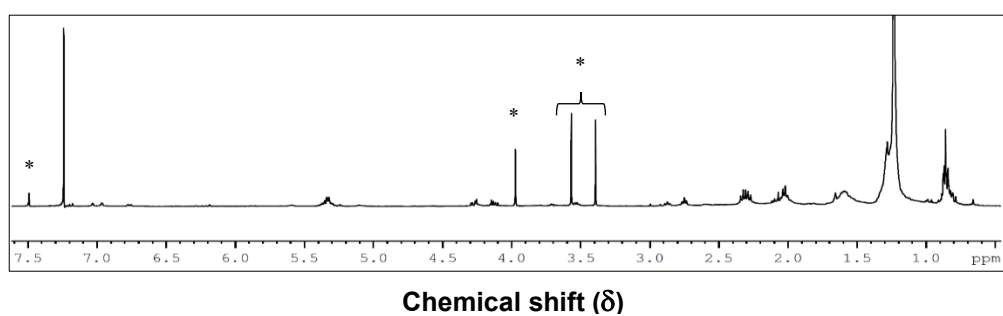


Figure S1. Husk <sup>1</sup>H NMR spectra of ethanol 2 hour extractions. Residual caffeine, observed by <sup>1</sup>H NMR at δ7.51, 4.00, 3.59 and 3.41 ppm. detected in the husk ethanol extraction spectrum

\*= peaks attributed to caffeine, according to Wishart, Knox <sup>[1]</sup>.

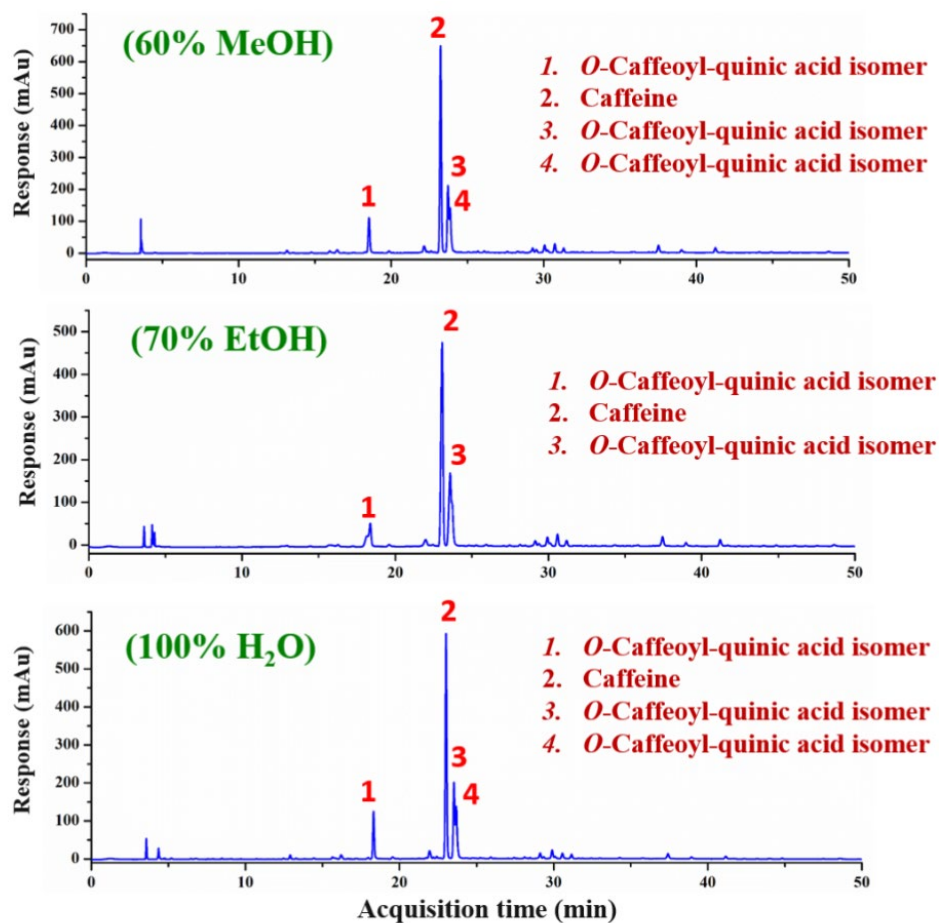
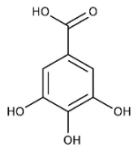
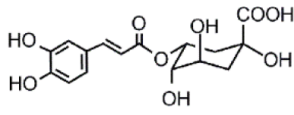


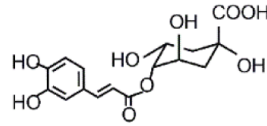
Figure S2. LC-MS chromatograms of SCGs ultrasonic bath extraction using different solvents for two-hours. The 3-4 major components are identified and it is likely the 4<sup>th</sup> peak associated with *O*-Caffeoyl-quinic acid isomer in the 70% EtOH spectra was present but not well resolved in the extract.



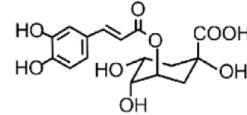
**gallic acid**



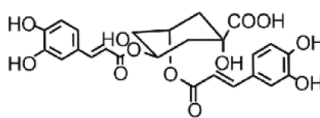
**3-caffeoylquinic acid**



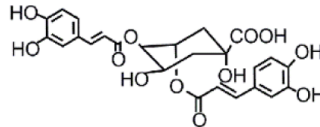
**4-caffeoylquinic acid**



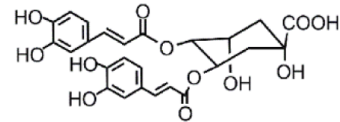
**5-caffeoylquinic acid**



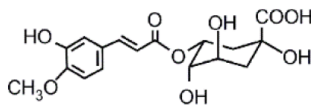
**3,5-diCaffeoylquinic acid**



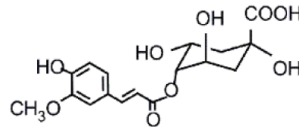
**3,4-diCaffeoylquinic acid**



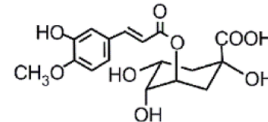
**4,5-diCaffeoylquinic acid**



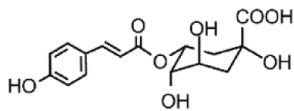
**3-feruloylquinic acid**



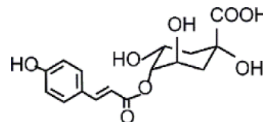
**4-feruloylquinic acid**



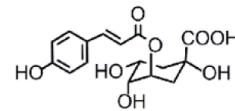
**5-feruloylquinic acid**



**3-p-coumaroylquinic acid**



**4-p-coumaroylquinic acid**



**5-p-coumaroylquinic acid**

Figure S3. Common Chlorogenic Acids found in Spent Coffee Grounds <sup>[2,3]</sup>

Table S1. Caffeine and Gallic Acid Equivalents (GAEs) concentrations from SCGs extractions with different solvents, averages  $\pm$  SD, in a ultrasonic bath.  $n=2$ . Letters represent the Tukey's post-hoc test ( $p= <0.05$ ), caffeine and GAEs were analysed separately. Caffeine was quantified by LC-MS and GAEs by colorimetric analysis.

<b>Solvent</b>	<b>Extraction time (hrs)</b>	<b>Caffeine (mg/g)</b>	<b>GAEs (mg/g)</b>
60% MeOH	1.5	4.9 $\pm$ 0.2 <sup>b</sup>	21.8 $\pm$ 0.2 <sup>b</sup>
	2	4.9 $\pm$ 0.3 <sup>b</sup>	22.2 $\pm$ 0.3 <sup>b</sup>
70% EtOH	1.5	4.9 $\pm$ 0.1 <sup>b</sup>	23.1 $\pm$ 0.8 <sup>b</sup>
	2	4.8 $\pm$ 0.1 <sup>b</sup>	22.3 $\pm$ 1.7 <sup>b</sup>
100% DI H <sub>2</sub> O	1.5	4.1 $\pm$ 0.1 <sup>a</sup>	17.0 $\pm$ 0.3 <sup>a</sup>
	2	4.1 $\pm$ 0.1 <sup>a</sup>	16.8 $\pm$ 0.1 <sup>a</sup>
<b>Solvent</b>	<b>Extraction time (hrs)</b>	<b>Caffeine (mg/g)</b>	<b>GAEs (mg/g)</b>
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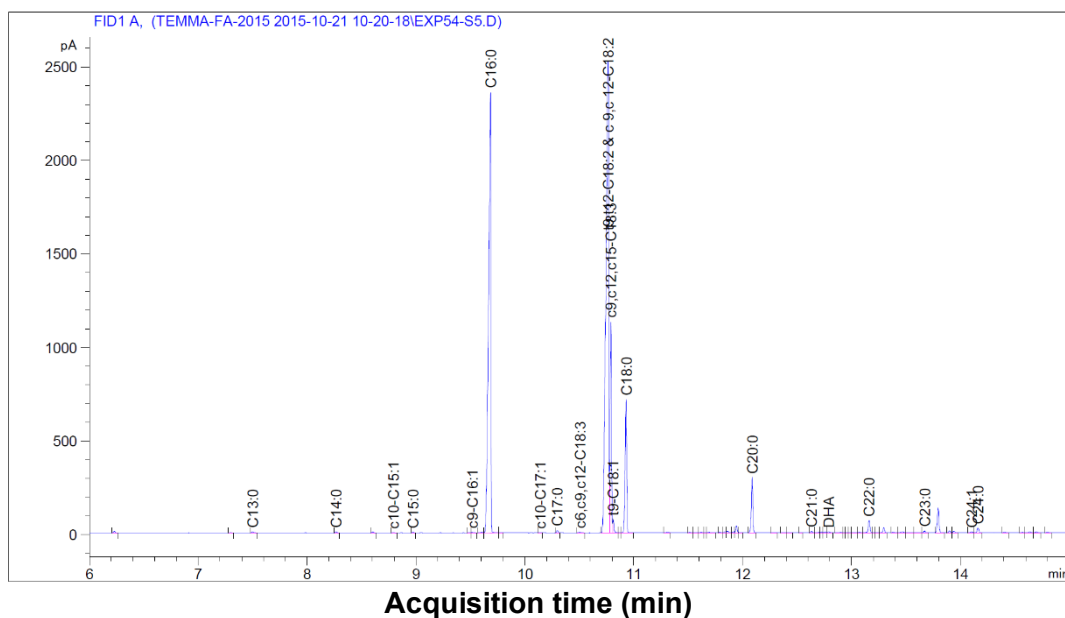


Figure S4. Typical GC chromatogram of SCGs (similar chromatograms were obtained for both ethanol and hexane extraction)

### References

- [1] D. S. Wishart, C. Knox, A. C. Guo, R. Eisner, N. Young, B. Gautam, HMDB: a knowledgebase for the human metabolome. *Nucleic acids research*. **2009**;37(Database issue):D603-10.
- [2] Daniele Del Rio 1, Angeliqe Stalmach 2, Luca Calani 1 and Alan Crozier, Bioavailability of Coffee Chlorogenic Acids and Green Tea Flavan-3-ols. *Nutrients* **2010**; 2: 820-833
- [3] H. Xu, W. Wang, X. Liu, F. Yuan, Y. Gao. Antioxidative phenolics obtained from spent coffee grounds (*Coffea arabica* L.) by subcritical water extraction. *Industrial Crops and Products*. **2015**;76:946-54.